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CITRUS
PROCESSING CONFERENCE

U. S. Fruit and Vegetable Products Laboratory
Winter Haven, Florida

September 22, 1960



LIST OF ATTENDANCE

Tenth Annual
CITRUS PROCESSING CONFERENCE
Florida Room Citrus Building
Winter Haven, Florida
September 22, 1960

UNITED STATES DEPARTMENT OF AGRICULTURE
Agricultural Research Service
Southern Utilization Research and Development Division
U.S. Fruit and Vegetable Products Laboratory

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PROGRAM AND ABSTRACTS OF PAPERS

TENTH CITRUS PROCESSING CONFERENCE

September 22, 1960

Florida Room, Citrus Building Winter Haven, Florida

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IN

TENTH CITRUS PROCESSING CONFERENCE

SOUTHERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION

U. S. Fruit and Vegetable Products Laboratory, Winter Haven, Florida U. S. Fruit and Vegetable Products Laboratory, Weslaco, Texas

WESTERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION

Fruit and Vegetable Chemistry Laboratory, Pasadena, California Western Regional Research Laboratory, Albany, California DISTRIBUTE CHIEF CONTRACTOR

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TENTE CIERLS PROCESURE CONFERENCE

SOUTHERN DIRECTOR RESERVED AND DEVELORED DIVISION

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PROGRAM

CITRUS PROCESSING CONFERENCE September 22, 1960

MORNING SESSION - 9:30 A. M.

(M. K. Veldhuis, In Charge, U. S. Fruit and Vegetable Products Laboratory, Winter Haven, Florida, Presiding)

OPENING REMARKS	. M. K. Veldhuis			Page
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Albert N. Western Re	PHYSIOLOGICAL EFFECTS Booth, M. Sid Masri an gional Research Labora ented by Albert N. Boo	d Dorothy J. Robbins tory, Albany, Califor		. 6
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AFTERNOON SESSION - 1:30 P. M.

Pa	age
RECENT DEVELOPMENTS IN FOAM-MAT DRYING	.0
PRELIMINARY INVESTIGATIONS WITH NEW FOAM-MAT DRYING UNIT AT WINTER HAVEN	2
DEMONSTRATION OF OPERATION OF EXPERIMENTAL FOAM-MAT DRYING UNIT AT THE U.S. FRUIT AND VEGETABLE PRODUCTS LABORATORY, 600 AVENUE S, N. W., WINTER HAVEN, FLORIDA	
Demonstration will begin at about 2:45 p. m. and continue to about 4:15 p. m.	
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FURTHER OBSERVATIONS ON THE STABILITY OF CHILLED CITRUS SALADS

N. B. RUSHING and V. J. SENN
U. S. Fruit and Vegetable Products Laboratory
Winter Haven, Florida

Studies on the stability of packs of chilled mixed grapefruit and orange sections were continued during the past year. Cover syrups containing two levels (0.033% and 0.066%) each of sodium benzoate, potassium sorbate, and a 1:1 mixture of these were dispensed into pint jars and the jars filled in a commercial plant by the regular plant personnel. Controls were put up in syrup without added preservative. After pre-chilling in the plant, the pack was divided for storage at 0°, 30°, 40°, and 50° F. Those intended for frozen storage were transferred to plain tin #2 cans and sealed.

Sampling was twice weekly at 50°, weekly at 40°, and biweekly at 30° F. The samples at 0° F. were used as controls for taste testing. Tasting was by the triangular method. Two jars were withdrawn from storage at the indicated intervals and the cover syrup sampled for microbiological and chemical examination. The residual syrup and sections were pooled, comminuted in a blendor, and submitted to the taste panel for organoleptic evaluation. Two cans containing a similar cover syrup were thawed, blended, and used as controls. The samples were considered to be spoiled when the panel found the experimental samples were inferior to the controls.

The panel found the experimental samples spoiled after one week at 50°, and 5 or 6 weeks at 40° F. Samples without preservative were judged spoiled after 12 weeks at 30° F. Storage life at the lower level of preservative was about 16 weeks at 30° F. Spoilage at the higher preservative level was not as definite at 16 weeks, at which time the supply of samples was exhausted.

A distinct drop in microbiological population was observed over several weeks at 30° F., followed by a definite rise over the balance of the storage period. Sodium benzoate more effectively inhibited microbial growth than did potassium sorbate. Plate counts could not be used as an indication of spoilage. Storage life was not increased markedly by any preservative at the levels tested. The panel was unable to detect the preservatives at the levels used.

5.1

PROGRESS REPORT ON TIME-TEMPERATURE-TOLERANCE STUDY OF FROZEN CONCENTRATED ORANGE JUICE

THEO J. KEW
U. S. Fruit and Vegetable Products Laboratory
Winter Haven, Florida

Time-temperature-tolerance investigations have been continued. New facilities have permitted examination of all packs by taste panels, and flavor stability data are becoming available. New information is being obtained on the effects of various storage treatments on the residual storage life of concentrates. There are three phases of this work involving regular commercial packs, experimental packs of 4-, 5 1/3-, and 6-fold concentration, and packs canned with and without vacuum or inert gas in the headspace.

Commercial packs of 4-fold (42° Brix) frozen concentrate are being studied for cloud and flavor stability after storage at 20°, 15°, 10°, and 5° F. When stored above zero, damage was sustained by these concentrates. A loss of subsequent storage life at household refrigerator temperature (40° F.) has been shown to result even though the time of such temperature exposures was such that no change was obvious upon immediate inspection of the concentrates. Storage life was measured by a cloud stability test and flavor. As long as there was no loss in cloud, there was no detectable flavor change. The panel often detected flavor damage when residual cloud life at 40° F. was just exhausted. The greater the time-temperature exposure above zero, the shorter the residual storage life with loss of cloud and flavor stability.

Experimental 4-fold (42° Brix), 5 1/3-fold (53.5° Brix), and 6-fold (58° Brix), concentrates made from a single lot of fruit have been compared for behavior on storage. Particular attention was paid to the effect of degree of concentration on flavor stability. Both cloud and flavor stability increased with increased concentration. The taste panel was able to detect a flavor difference between portions stored at 40° and 0° F. for three series of 4-, 5 1/3-, and 6-fold concentrates coded M, N, O; Q, R, S; and W, X, Y, at M=8, N=14, O=12, Q=2, R=6, S=6, and W=2, X=3, Y=5 weeks.

The average results showed a shorter storage life for the 42° Brix concentrates but no difference between the 5 1/3- and 6-fold products. An effort was made to ascertain whether the taste panel preferred the 4-fold or either of the two more concentrated products after storage at 0° F. for one year. The panel was unable to detect a difference in flavor in either case and it was concluded there was no basis for a preference.

Experimental 4-fold concentrates sealed in air, nitrogen, and under vacuum are being subjected to storage and then examined for cloud and flavor stability. This last category of experiments has been introduced as a preliminary study to determine the effect of oxygen on the quality characteristics of frozen concentrated orange juice.



STUDIES ON THE FLAVONOID COMPOSITION OF GRAPEFRUIT

ROBERT M. HOROWITZ AND BRUNO GENTILI
Fruit and Vegetable Chemistry Laboratory
Pasadena, California

Presented by
ALBERT N. BOOTH
Western Regional Research Laboratory
Albany, California

A study has been made of the flavonoid composition of Marsh Seedless grapefruit collected at various stages of growth. In all samples the principal flavonoid compound was naringin. A second flavonoid, previously unreported in grapefruit, was found to be poncirin. Poncirin had been discovered earlier by Hattori in the trifoliate orange (Poncirus trifoliata) and had been shown by him to be the glycoside of naringenin 4'-methyl ether. Since poncirin is extremely bitter (its bitterness is comparable to that of naringin) it seemed probable that the rhamnoglucose portion of it must be in the form of nechesperidose, rather than rutinose. (Last year it was reported here that bitter flavanone glycosides contain neohesperidose, while the nonbitter ones contain rutinose.) It has now been confirmed that the poncirin of grapefruit is the neohesperidose of naringenin 4'-methyl ether. Analysis of the various samples by a specially developed spectral method showed that the ratio of poncirin to naringin was about 1:5, regardless of the time of year that the fruit was collected.

Another phenolic glycoside which we have isolated from grapefruit (and have shown to be present in lemons and oranges) is phloroglucinol beta-D-glucoside ("phlorin"). Phlorin had not been previously recognized as a natural constituent of plants. The isolation of phlorin from citrus is of interest since (a) it would seem to constitute a plausible substrate for browning reactions, and (b) it is reported to produce glucosuria in laboratory animals, though it is far less potent than phloridzin in this respect. We have found that the addition of a small quantity of phlorin to lemon juice enhances the rate of darkening on long standing.

Preliminary experiments on the flavonoid composition of Valencia oranges have been carried out. These will be reported briefly.

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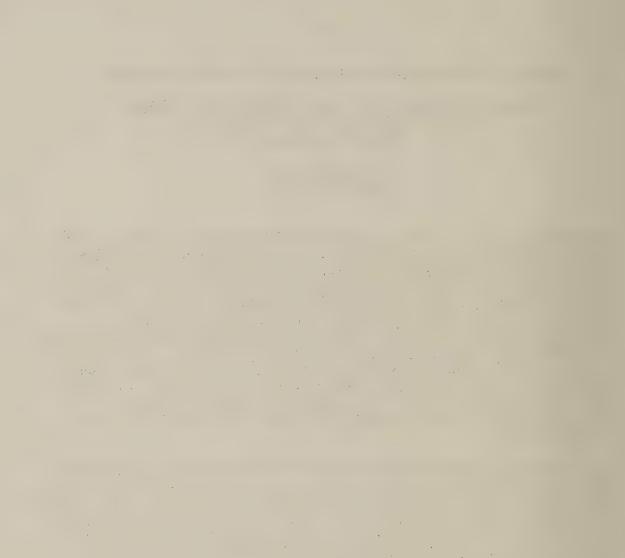
STUDIES ON THE PHYSIOLOGICAL EFFECTS OF CITRUS FLAVONOIDS

ALBERT N. BOOTH, M. SID MASRI AND DOROTHY J. ROBBINS Western Regional Research Laboratory Albany. California

Presented by ALBERT N. BOOTH

We have now clearly established that the flavonoids in citrus are not only absorbed from the gastrointestinal tract after oral ingestion, but in addition are capable of exerting physiological effects in the animal body after which they are excreted in the urine in the form of breakdown products. Thus, m-hydroxyphenylpropionic acid is the major metabolite excreted when hesperidin, diosmin and eriodictyol are ingested by rats and rabbits. The major metabolite formed from naringin is p-hydroxyphenylpropionic acid. Obviously, these C6 - C3 compounds arise from the right-hand or B ring of the flavonoid molecule. Until recently the fate of the left-hand or A ring was unknown. It has now been established that the A ring undergoes cleavage followed by beta-oxidation of three two-carbon fragments which are then further oxidized to CO2.

The clinical use of flavonoids provides evidence that these substances may exert physiological effects in man. Numerous physiological effects of flavonoids have been reported in experimental animals. Until recently the primary mechanism of action of a flavonoid in the animal body was attributed to the antioxidant effects on circulating epinephrine and ascorbic acid. A new action mechanism involving stimulation of the pituitary-adrenal axis was discovered recently by Masri and DeEds. This aspirin or salicylic acid-like effect as measured by involution of the thymus gland provides a more logical explanation for many of the seeminly unrelated physiological effects associated with the flavonoids.



RESEARCH ON THE ALDEHYDES AND HYDROCARBONS OF CITRUS OILS

W. L. STANLEY, R. M. IKEDA, S. H. VANNIER AND L. A. ROLLE Fruit and Vegetable Chemistry Laboratory Pasadena, California

Presented by R. M. IKEDA

The following hydrocarbons have been isolated and identified in lemon oils: α -pinene, β -pinene, sabinene, myrcene, d-limonene, %-terpinene, terpinolene and p-cymene. Identification of these compounds was based on comparison of gas-liquid chromatography (GLC) retention times and infrared spectra. With the exceptions of sabinene and myrcene, identification of these compounds was further verified by nuclear magnetic resonance spectra. A ninth hydrocarbon was tentatively identified as α -thujene.

Samples of domestic lemon oils accumulated from 1955 to the present time were analyzed for hydrocarbon content by downward elution on chromatostrips followed by gas-liquid chromatography. Wide variations were observed for the relative concentrations of β -pinene and d-limonene in these samples. The optical rotations of the oil samples were found to be directly proportional to the d-limonene content and inversely proportional to the β -pinene content. These phenomena explain the reason for the wide natural variation in optical rotation of lemon oils and provide a convenient means for detecting the addition of partially racemized d-limonene to lemon oils.

Lemon oil samples which had been stored in loosely-capped bottles for several months were found to contain higher amounts of p-cymene than freshly prepared and well sealed stored samples. Samples high in p-cymene were low in x-terpinene. Evidence was obtained which conclusively demonstrated that x-terpinene is the major precursor of p-cymene formed during deterioration of lemon oils. p-Cymene is responsible for the so-called "cymie" off-flavor that develops in oxidized oils.

The major hydrocarbons in grapefruit and orange oils were found to be di-limonene, myrcene and α -pinene (d-limonene represented approximately 98% of the hydrocarbon mixtures in both oils). Traces of α -thujene, sabinene, α -phellandrene, γ -terpinene and p-cymene were also found in orange oils. Identifications were based on comparison of GLC retention times. The relative abundance of the hydrocarbons in lime oils resembled those in lemon oils.

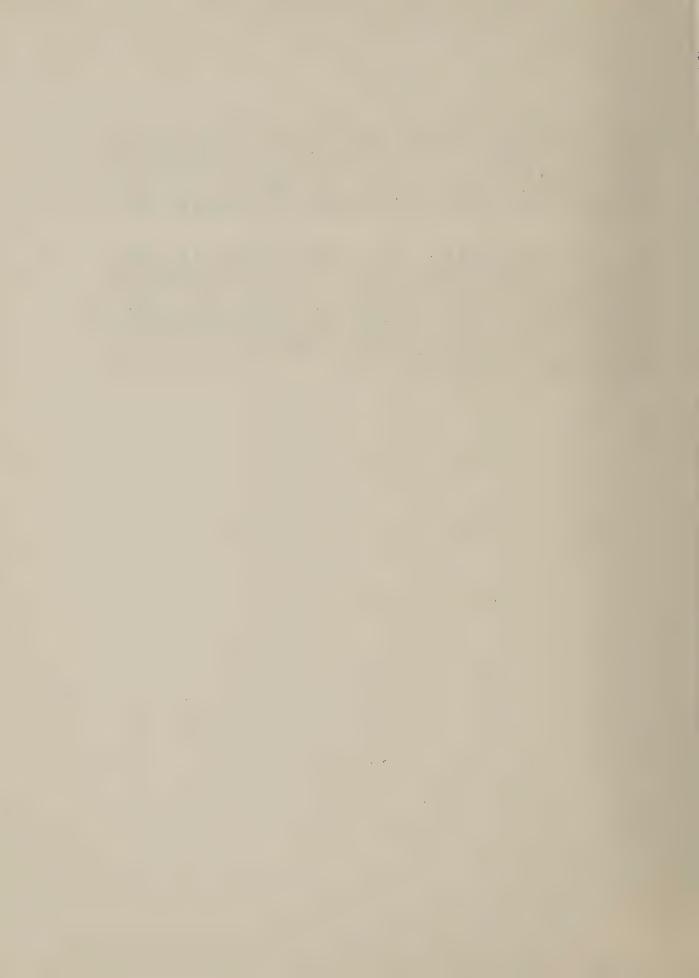
The major aldehydes in lemon oil were determined by their conversion to water-soluble Girard derivatives, regeneration with formaldehyde, followed by gas-liquid chromatography. Only the amounts of n-octanal,

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n-nonanal, n-decanal, n-undecanal, n-dodecanal, citronellal and citral were considered in this survey. The ratio n-nonanal/n-octanal appeared to be related to fruit growing area. The relative amount of citral present in the aldehyde mixture also appeared to be related to growing areas and to optical rotation. The relationship of citral to rotation may provide a means for determining the addition of citral to lemon oils.

Lime oil resembled lemon oil in the relative abundance of the major aldehydes but contained higher amounts of n-decanal and n-dodecanal. Orange oils and grapefruit oils were similar. These oils in contrast to lemon oil, were quite low in n-nonanal. Navel orange oils contained more citral than Valencia oils. Chief variations in the major components were in the amounts of n-octanal and n-decanal. The detailed chromatograms of the minor aldehydes were much more complicated for orange oil than for lemon oil.



WHAT DO CONSUMER PREFERENCE TESTS OF CANNED JUICE FROM RED GRAPEFRUIT INDICATE?

BRUCE J. LIME
U. S. Fruit and Vegetable Products Laboratory
Weslaco, Texas

The U. S. Fruit and Vegetable Products Laboratory, Weslaco, Texas, Texas A&M College, and the Agricultural Marketing Service, USDA, made cooperative studies of consumer preferences for white and red grapefruit juice, both pulp-fortified and unfortified.

One test was conducted in two areas (Dallas and Houston, Texas) where red grapefruit and juices were familiar products; and the other test was conducted in an area (Des Moines, Iowa) relatively unfamilar with red meated grapefruit. In the familiar area there was a preference for canned red grapefruit juice over canned white grapefruit juice. The reason given for this preference was that the red juice was sweeter, although chemically the red juice showed no difference in sweetness and a blind taste panel could not distinguish the difference between them. This would indicate that in these areas where red grapefruit was familiar there was an association of redness with sweetness.

In the area where red grapefruit was not familiar there was a slight preference for juice from white grapefruit. Pulp-fortification had no effect on the preferences shown for the juices.

The results were based on limited observations in three areas, using grapefruit juices of acceptable quality but not the highest quality that could be commercially manufactured.

RECENT DEVELOPMENTS IN FOAM-MAT DRYING

ARTHUR I. MORGAN, JR., AND R. P. GRAHAM Western Regional Research Laboratory Albany, California

Presented by ARTHUR I. MORGAN, JR.

Foam-mat drying is a new method of producing instant food powders by air drying stable foams. These foams dry rapidly because of capillary transport of water to a free surface from inside the foam mass. Rapid drying results in high product quality. The dry foam structure promotes instant rehydration of the product. The very low levels of product moisture which are possible are helpful for storage stability.

Many liquid foods, including citrus juice concentrates, can be converted to stable foams by incorporating a small proportion of an edible foam stabilizer. In the case of orange juice, an amount of glyceryl monostearate equal to 1% of the orange solids can be used with good results. Another foam stabilizer, soluble soya protein with methocel, is also useful as an alternative. In either case, a good stiff foam can be produced by passing the food and stabilizer together with 2-1/2 volumes of gas (air or nitrogen) through a high shear mixer.

The foams have been air dried in thin layers or extruded pieces lying on a flexible moving surface. For this method, about 0.1 lb. of product per hour per square foot of installed area can be expected. Drying times depend on air temperature and piece thickness. The warmest air practicable with this method is about 160° F. At this temperature, using 1/8" pieces, drying times are 15 to 20 minutes. This method is satisfactory for pilot-plant use and for larger scale if the right equipment could be designed. It is not as fast or as efficient as could be desired. We have therefore begun to develop other techniques.

The crater technique is a way of extending stable foams in the direction of drying air flow in such a way that the average distance to a free surface from within the foam is a minimum. The foam is first extruded as a thin layer onto a perforated metal sheet. The sheet with its load of foam moves over an air blast which pierces the foam above each perforation. The foam is spread aside but not blown off the sheet. This pierced or cratered formation is retained because of the stiffness of the foam. The drying air is them passed through the perforations until the foam is dry. The cratered layer of dry foam can then be cooled. It can easily be scraped off the sheet and packaged. In the case of citrus products, the cooling and packaging must be done in dehumidified air, although the drying air itself need



not be dehumidified. With this technique, about 0.5 lb: of product per hour can be obtained from each square foot of installed surface. Furthermore, the surfaces are more durable and can be more conveniently arranged in an air stream than the flexible surfaces used with the thin layers or extruded pieces.

Design of dryers for foam-mat drying is under study. The best results are obtained by staging the drying air. The warmest and dryest air should contact the freshest foam. If the foam and air move in the same direction in the first stage, the most even product temperature will be maintained and surface over-drying will be avoided. About 75% of the moisture in a foam could be removed by pausing a minute in each of three positions in a concurrent first stage, the air being introduced at 200° F. The remaining moisture should be removed in a countercurrent second stage so that very dry product is obtained. Such a second stage might require the trays to pause a minute in each of six positions, the air being introduced at 130° F. This stage might profitably be divided into two sections with the first having a warmer air supply than the second.

These concepts are being used in design of a small scale fully automatic and continuous dryer at Albany, California. The stages will be arranged separately as vertical stacks. Elsewhere, on a small commercial scale, plans are being made to use 4 ft. square stainless steel sheets in a single vertical stack, the first stage air entering at the bottom. This concept can be further scaled up in several ways. One such idea involves a square helix of trays winding up through the air stream. These concepts are under study at the present time.

PRELIMINARY INVESTIGATIONS WITH NEW FOAM-MAT DRYING UNIT AT WINTER HAVEN

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A cooperative investigation has been undertaken by the Florida Citrus Commission; the Western Utilization Research and Development Division, Albany, California; and the U. S. Fruit and Vegetable Products Laboratory, Winter Haven, on the production of powdered orange juice by a new method. This new method called Foam-mat drying has been developed for fluid food products at the Western Utilization Research and Development Division. At the 1959 Citrus Processing Conference Dr. W. D. Ramage of WURDD discussed the method and presented a sample of powdered orange juice for tasting. The method is unique in that drying is accomplished in a blast of hot air (ca. 165° F.) under normal atmospheric pressure. In order to permit rapid drying, concentrated orange juice is whipped into a stable foam which forms a porous structure during drying. A small amount of an edible foam stabilizer such as saturated mono-glyceride from dehydrated lard is added.

A new Foam-mat drying unit has been installed at the U. S. Fruit and Vegetable Products Laboratory as a result of the interest noted at the 1959 conference and subsequently. This new unit has been designed by engineers at the Western Utilization Research and Development Division based on their experience with the previous model. The Florida Citrus Commission has purchased and installed the drier and auxiliary equipment, and purchased some of the supplies. The equipment has been undergoing "shakedown" runs and adjustments so that it can be operated under controlled conditions. A few preliminary runs have been made and these will be discussed. Indications are that if the method is conducted properly very little, if any, damage is done to the product. Sometimes a little heated flavor has been noted in the material directly from the drier, but when flavored with "locked-in" oil, it is no longer noticed.

Extensive investigations in cooperation with the Florida Citrus Commission and the Western Division are planned to determine the effects of variations in drying temperatures, in drying time, in belt loading, and other factors on quality of the product. Work will also be undertaken on the effects of various factors such as the effect of moisture content of the final product, "in package" desiccants, inert gas packaging, and other factors on stability in storage.

DEMONSTRATION OF OPERATION OF EXPERIMENTAL FOAM-MAT DRYING UNIT AT WINTER HAVEN

The demonstration will be conducted at the U.S. Fruit and Vegetable Products Laboratory, 600 Avenue S, N.W. The equipment will be in operation for an hour and a half. This will give everyone an opportunity to watch each stage of the operation and talk with personnel about the process.

The process starts with the preparation of a stabilizer foam by mixing the mono-glyceride with hot water in a blendor. The resulting product looks much like foamy shaving cream. Some of this stabilizer foam is mixed with concentrated orange juice (42° to 65° Brix) and mixed with a wire whip for about ten minutes until a stiff light yellow foam is produced.

The foamed concentrate is extruded in strips about the diameter of coarse spaghetti onto the surface of a moving fibre glass-teflon belt which passes through successive stages in the drier where hot air is blown across the surface of the belt. Drying times currently used are from 12 to 15 minutes. The product then passes into a cooling section in which room temperature air is blown across the belt.

The belt finally passes into the low-humidity room where the relative humidity is maintained at 15% or below. Cool air from a room air conditioner is used to further cool the product and harden it so it strips easily from the belt as it passes over the take-off roll.

Loosened pieces of dried orange juice tend to adhere to the belt because of a static charge. A plastic doctor blade strips the loose pieces from the belt.

The dried orange juice is bulky as it comes from the drier, and its bulk must be decreased before packaging. The product is ground in a small grist mill, compressed in a hydraulic press and ground again in the same mill. It is them ready for packaging. Equipment is at hand for packing under vacuum, under inert gas, or with air.



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L. Jurd and L. A. Rolle

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